# EXTRACTION OF SUGAR

FROM

# DRIED SUGAR BEETS

BY

R. F. SHERMAN I. Z. ETTENSON

W. W. PATRICK

Armour Institute of Technology

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dried sugar beets

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# A THESIS

PRESENTED BY

R. F. SHERMAN  I. Z. ETTENSON  W. W. PATRICK

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# THE EXTRACTION OF SUGAR

# FROM

# DRIED SUGAR BEETS

R. F. SHERMAN   I. Z. ETTENSON   W. W. PATRICK

* * *

## EXTRACTION OF SUGAR FROM DRIED BEETS.

To understand the advantages and disadvantages of this process, it will be necessary to compare it with the fresh beet process which is now used in practically all plants. Briefly, the fresh beet process is as follows: The beets are washed to remove adhering dirt, and cut into slices called cossettes. A cossette is about the size of a pencil, 3 or 4 inches long, and has a V shaped cross section. This shape gives it the maximum strength and surface. These cossettes are then placed in large cast iron cells called diffusors, and treated with hot water. An average factory is provided with a series of from 10 to 15 of these cells, known as the diffusion battery. The water passes through each cell in the series, entering the cell containing beets nearest to exhaustion and leaving through the cell containing freshly charged beets. By this system juice of maximum concentration is obtained, and the sugar is completely extracted from the beet. The action of the water in this process may be put under two headings:-

(1) In the case of the beet cells which have been broken in cutting, the action is simply lixiviation; that is, the dissolving out of the contents of the ruptured cells.

(2) Where the cell walls have not been broken, the action becomes osmotic, and since crystalline substances diffuse

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by osmosis more rapidly than albuminous substances, the result of this action is the obtaining of a sugar solution of higher purity than that in the original beet cell. On the other hand, the solution obtained by lixiviation is of identical purity with the juice of the original beet. This point will be referred to later in the description of the dried beet process.

The purification, concentration and crystallization of the juice from the diffusion battery, are points which will be discussed later.

The advantages and disadvantages of the fresh beet process may be summarized as follows:

ADVANTAGES:-

(a) A juice may be obtained which is of higher purity than in the original beet.

(b) The juice obtained cannot be of higher concentration than in the beets themselves. This constitutes an advantage in a certain sense, for thin juice is easily handled by means of pumps, can be easily filtered, and for several reasons sugar losses are reduced.

(c) The growth of bacteria is not so rapid in thin as in more concentrated juice.

(d) The vicosity of thin juice being low, the rapidity of circulation in the battery is increased, thus raising its capacity.

DISADVANTAGES:-

(a) Beets under favorable conditions will seldom keep

longer than 3 or 4 months, thus limiting the period of operation of a plant to but a short time in the year, usually from September 15 to January 15. This necessitates the plant lying idle the remainder of the year, during which time interest and depreciation charges accumulate. This constitutes the most serious drawback to the fresh beet process as now in operation.

(b) With thin juice the cost of evaporation necessary for crystallization is high, constituting one of the greatest expenses in thisprocess.

(c) The weight and bulk of the fresh beets is another drawback. It has been found by experience that a plant to be successful must handle at least 200 tons of beets per day, and the cost of a plant of even this capacity requires a large amount of capital. Plants are now in operation which handle 2,000 tons of beets per day. Almost as great a disadvantage due to the weight and bulk of the beets is encountered in the transportation problem.

From the above considerations it will be seen that the principal objections to the old process are:-

(1) - The shortness of the beet sugar campaign.

(2) - The initial cost of the plant, and the interest and depreciation charges while it is idle.

It will now be my endeavor to show that a process involving the use of dried beets will overcome these difficulties to a great extent, and at the same time introduce advantages not mentioned above.

The process of preparing dried beets (as patented) is

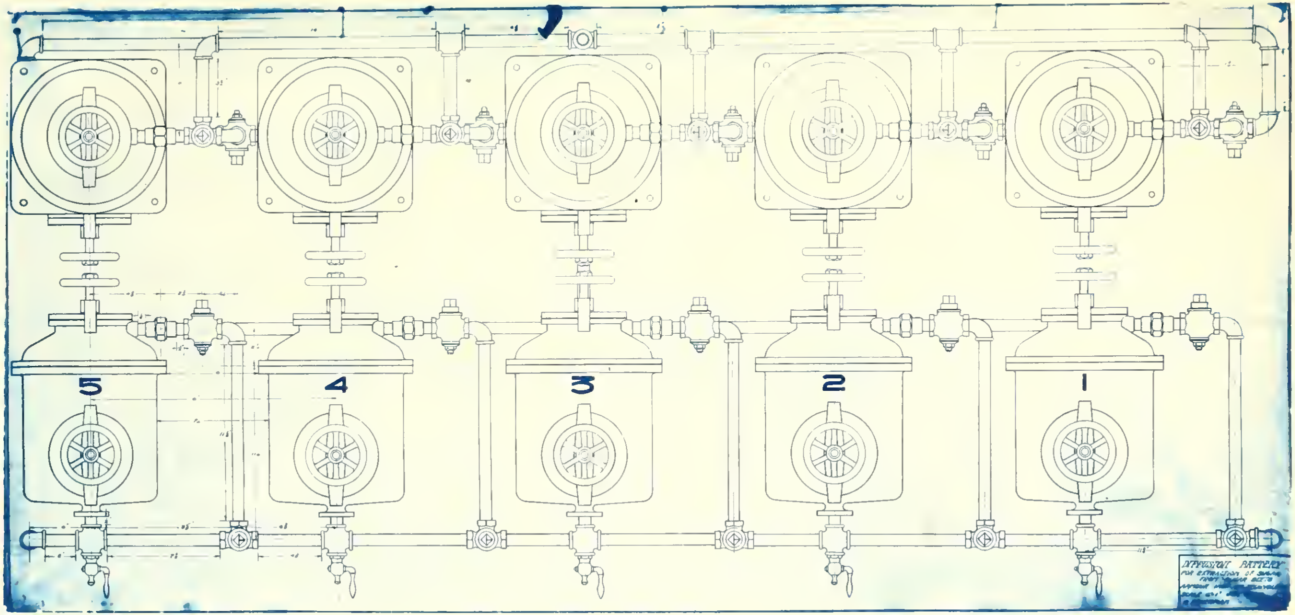
as follows: Fresh beets are cut into cossettes identical with those used in the fresh beet process. These are placed on endless belts, and dried in steam-heated kilns, the moisture content being reduced from about 77% to about 1% on the original beet (this corresponds to 3 or 4% on the dried beet). The dried cossettes are then ground between corrugated rolls to about the size of wheat, in which condition it can be stored indefinitely without undergoing any change of importance.

The cost of drying beets by this process under favorable conditions may be as low as 15 cents per ton. The most economical system for drying beets would be to have the kilns located in the beet raising district where the drying would be done, after which the dried beets could be shipped to the factory. This would greatly reduce the cost of transportation. Since the dried beets can be stored indefinitely, a plant operating on this process could be run the entire year, and since the weight and bulk of the beets have been reduced about 75%, the initial costs of a plant of a given capacity per year would be decidedly less than the cost of a plant using the fresh beet process.

The question now is,-- Can sugar be extracted from dried beets by an economical process. This question has been partially answered by a series of experiments with dried beets carried on at the Armour Institute of Technology, Chicago.

## Results of Experiments with Dried Beets.

Apparatus:- This consisted of a battery of five cells or diffusors constructed on the principle of diffusors now in


DIFFUSION BATTERY
FOR EXTRACTION OF SUGAR
FROM SUGAR BEETS

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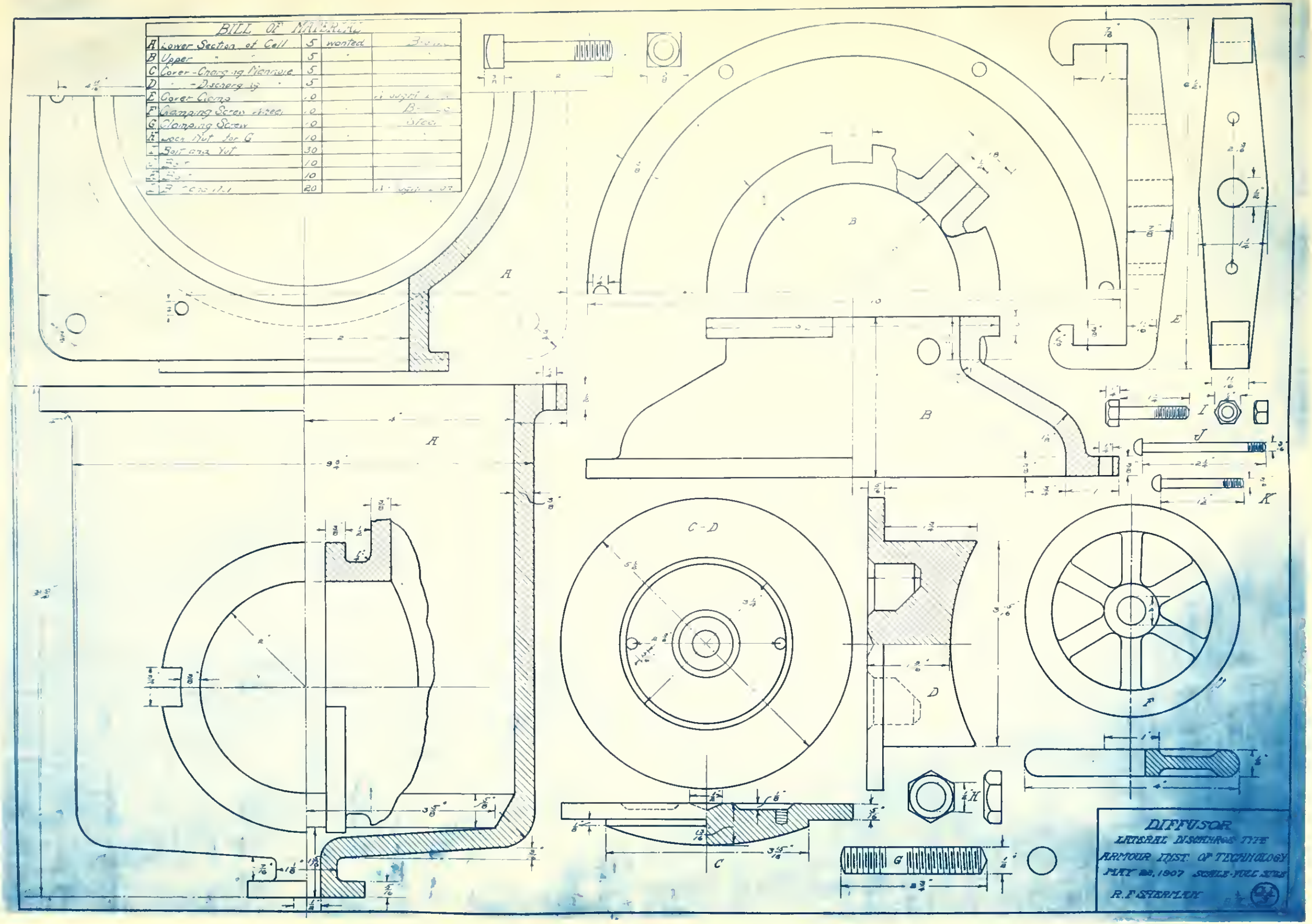

| BILL OF MATERIAL | | | | |
|---|---|---|---|---|
| A | Lower Section of Cell | 5 | wanted | [illegible] |
| B | Upper " | 5 | " | |
| C | Cover-Charging Manhole | 5 | | |
| D | " -Discharging " | 5 | | |
| E | Cover Clamp | 10 | | [illegible] |
| F | Clamping Screw Sleeve | 10 | " | [illegible] |
| G | Clamping Screw | 10 | | Steel |
| H | Lock Nut for G | 10 | " | |
| I | Bolt and Nut | 30 | | |
| [illegible] | Bolt | 10 | | |
| [illegible] | Bolt | 10 | | |
| [illegible] | [illegible] | 20 | | [illegible] |

DIFFUSOR
LATERAL DISCHARGE TYPE
ARMOUR INST. OF TECHNOLOGY
MAY 28, 1907 SCALE-FULL SIZE
R. F. SHERMAN

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actual operation. Certain changes were, however, necessary on account of the difference in general character between dried beets and cossettes. The accompanying detail and assembly drawingsare self explaining. The system of piping employed is similar to that used in all modern factories, with the exception that means were not provided for using warm water in the battery. The beets in the cell rest on a 40 mesh brass gauze.

The method of operation of the battery is as follows: Cell #1 is first filled about two-thirds full of water, which is run in from the bottom. A charge of four pounds of dried beets is now poured in through the upper manhole and the mixture is stirred a few times, or until uniform. The upper manhole cover is now clamped in position and more water is sent in at bottom of cell until the air is completely forced out, then the flow is reversed. On reversing, the partly saturated juice from Cell #1 passes into cell #2, where it is used in "mashing" a fresh charge of beets. It was first attempted to use only the juice from a preceding cell in "mashing" the contents of a fresh charged cell, but the concentration became so great as to interfere with circulation. We therefore diluted this juice with about 25% of fresh water, which greatly improved the working of the battery. Cells #3, #4, and #5 are each filled in turn, always from the bottom to drive out the air, after which the flow is reversed. As soon as cell #5 is filled and "mashed" the stop cock at its lower end is opened and juice drawn off. The following conditions now exist in the battery: Water is entering at the top

of cell #1, and after passing down goes to the top of #2. In a similar manner it passes through cells #3, 4 and 5, gradually becoming more concentrated. Since cell #1 is being treated with fresh water it will become exhausted first. It is then cut out of the battery, discharged, recharged and becomes the last cell in the series. In this way the process becomes practically continuous.

In the short time at our disposal the results obtained were elementary, but they show quite conclusively that the process can not only compete with the fresh beet process, but show several distinct advantages over the latter, namely,-

(1) The charging of the cells with dried beets can be done very rapidly as a comparatively small bulk of beets will completely fill a cell when they have been allowed to stand a minute or two in contact with water.

(2) The particles being much finer than in the cossette process the water comes in more intimate contact with the beets, thus decreasing the time required for complete extraction.

(3) The sugar content of the dried beet being in the neighborhood of 60%, a juice of almost any concentration can be obtained. The obtaining of a concentrated juice means a great saving in the cost of evaporation.

(4) Since in grinding dried beets a great many of the cells are broken, the extraction process becomes almost entirely one of lixiviation rather than osmosis. Whether this

is an advantage or a disadvantage remains to be demonstrated.

(5) The effect of drying beets on the protein which they contain is important from the standpoint of using the exhausted pulp in making cattle feed. Samples of completely exhausted dried **beet** pulp showed 9.86% protein, while a sample not entirely exhausted giving a liquor of .2 Brix showed a protein of 10.27%.

(6) The exhausted pulp as it comes from the diffusor contains about 94% water and 6% dry substance. Of this moisture 60% to 70% can be removed by pressing, while with exhausted cossettes presses work very unsatisfactorily.

Results of Experiments with Dried Beets.

Analysis of Dried Beets.

| | |
|---|---|
| Moisture | -3.94% |
| Sucrose | -61.00% |
| Protein | 7.70% |
| Ash | 3.50% |
| Ether Extract | .46% |
| Starch | 5.85% |
| Crude Fiber | 4.00% |
| Undetermined (non sugars) | 13.55% |
| | 100.00% |

| | |
|---|---|
| Water Sol (Cold Water) | 82.45% |
| Water Sol (Hot " ) | 84.05% |

Protein in Exhausted Pulp.

| | |
|---|---|
| Extracted to .0 Brix | 9.80% |
| " " .2 " | 10.27% |

Exact data on the time required to completely exhaust a cell full of dried beets was difficult to determine, since it depends to a great extent on varying conditions, such as, the temperature and pressure of the water and the rapidity of circulation. A few results are as follows:

May 1, 1908.

| Cell | #1 | #2 | #3 | #4 | #5 |
|---|---|---|---|---|---|
| Charged | 9.00 | 9.07 | 9.16 | 9.31 | 10.00 |
| Exhausted | 10.15 | 11.20 | 12.05 | 1.16 | 2.40 |
| Time | 1.15 | 2.13 | 2.49 | 3.45 | 4.40 |
| Recharged | 10.42 | 11.30 | 12.20 | 1.25 | 3.05 |

The above data cannot be compared with similar data from a run with fresh beets, since with dried beetsthe amount of beets treated per volume of cell, and the concentration of the juice obtained, is much greater. For example, at 1:16 P. M. 42# of juice had been drawn off, which gave a Brix of 24.2 and a purity of 80.8%. This would be equivalent to about 84# of 12° Brix juice as would be obtained when using fresh beets. The additional advantage of having a juice of high concentration must be taken into account.

May 12, '08.

| Cell | #1 | #2 | #3 | #4 | #5 |
|---|---|---|---|---|---|
| Charged | 2.11 | 2.13 | 2.16 | 2.24 | 2.33 |
| Exhausted | 3.00 | 3.50 | 4.25 | -- | -- |

In above run drawing off began at 2:34 P. M. and the battery was shut down at 4:30, making the run practically two hours. In this time 62# of 18.6° Brix juice was drawn off which had a purity of 81.2%. This corresponds to about 93# of a 12° Brix liquor.

It willbe noticed here that cell #1 became exhausted in less than one hour, from which it would appear that with a properly designed battery and sufficient water pressure, this condition could be maintained throughout the entire run. In fresh beet practice the time required to exhaust a cell varies from 1-1/4 hrs. to 2 hrs.

May 20, '08.

| Cell | #1 | #2 | #3 | #4 | #5 |
|---|---|---|---|---|---|
| Charged | 10.00 | 10.04 | 10.13 | 10.22 | 10.28 |
| Exhausted | 11.14 | 11.48 | 12.18 | -- | -- -- |

74# of 16.6° Brix juice was obtained, corresponding to about 96# of 12° Brix juice. Time required to draw off 74# of juice was 1 hr. 50 min.

Data for Exhaustion Curve. Cell #1

Cell charged at 10.00 A. M.

| Time | Brix | Time | Brix |
|---|---|---|---|
| 10.08 | 7.0 | 10.42 | 1.0 |
| 10.12 | 6.0 | 10.46 | .8 |
| 10.16 | 4.5 | 10.50 | .6 |
| 10.22 | 4.0 | 10.54 | .5 |
| 10.26 | 2.8 | 10.58 | .4 |
| 10.30 | 2.1 | 11.02 | .3 |
| 10.34 | 1.7 | 11.06 | .3 |
| 10.38 | 1.1 | 11.10 | .3 |

A glance at these figures shows that extraction is very rapid at first, but gradually becoming less as the beets become exhausted. From this it would appear that extraction should not be carried too far at the expense of time. By not attempting to completely exhaust the beets, a great saving in time is accomplished, and at the same time the residual sugar does not represent a complete loss, since it enriches the pulp as a cattle food, bringing a correspondengly higher price.

Another point of interest in this process is the fact that at all degrees of exhaustion of the beets the purity of the juice remains constant at about 80%. The only possible explanation in my opinion is that the action of water on dried beets is entirely one of lixiviation due to the fact that in drying and grinding the beets the walls of the cells are ruptured.

Purity Data.

| Observed Brix | Corrected Brix | Purity |
|---|---|---|
| 1.20 | 1.20 | 83.3 |
| 1.50 | 1.50 | 83.3 |
| 4.75 | 4.75 | 82.1 |
| 19.05 | 19.45 | 81.2 |
| 24.20 | 24.40 | 80.8 |

In consulting the above data on the extraction of sugar from dried beets, it must be remembered that in every case cold water was used. There would no doubt be many advantages in the use of hot water, but owing to lack of time it was impossible to obtain any data on this method. In

closing this part of the discussion it may be said that the dried beet process undoubtedly offers decided advantages over the older process, the chief requirement being the designing of apparatus suitable for use with dried beets.

# DEFECATION AND CARBONATION.

The treatment of the juice as it comes from the diffusion battery is probably the most important part in the manufacture of sugar from sugar beets. Here is where it is determined whether the manufacture of the sugar will be a financial failure or success. Unless all labor saving devices are used and unless all other precautions are taken to insure a clear, pure, and nearly neutral juice for the concentrators, all kinds of difficulties will arise which will make the manufacture of sugar from beets a commercial failure. For instance, in the first liming of the raw juice, the lime must be added in just the right proportion; the right temperature must be attained; and the right length of time taken, or else a slimy juice will be the result and cost a lot of labor and time in filter-pressing. Or again, in the carbonation, just the right alkalinity must be attained, and over carbonation avoided, or invert sugar will result, causing a loss in sugar value. Thus it can be seen that an experienced man is needed to oversee these vital operations, a man who is not only practical, but one who has a theoretical knowledge of the chemistry involved in sugar manufacture.

With these few preliminary remarks I will try to explain in detail the various operations through which the raw juice must pass after it leaves the diffusion battery, and will try to show under what conditions the best possible

juice may be obtained, to be sent to the concentrators.

## First Liming.

The raw juices coming from the diffusor contain all the substances which were dissolved in the beet, and which have been extracted by diffusion. For 100 parts of sugar, there are 2-2-1/2 parts of albumen; 2-1/2-3 parts of other nitrogenous matter; 1/2-1 part reducing sugar; 1 part pentosaus, and .4-.6 oxalic acid. The inorganic constituents are chiefly potash, soda, lime, magnesia, phosphoric acid, sulphuric acid, chlorine, and small amounts of other acids and bases. The juice always has an acid reaction, the acidity being about 2 cc. of normal acid and 100 cc, juice.

By liming or defecating both suspended matter and most of the non sugars are removed from the juice. The lime acts both mechanically and chemically upon the impurities contained in the raw juice. It acts chemically in precipitating and decomposing the non sugars; and mechanically in carrying down the suspended matter with the precipitates. Chemically, the lime neutralizes the free acids and acid salts, and forms insoluble salts of the organic and inorganic acids, principally phosphoric and oxalic acids, these substances being insoluble in the alkaline juice. Lime, therefore, acts as a purifying agent, and changes the nature of those non sugars which it does not remove directly, so that favorable conditions are obtained for further working of the juices.

The best way to add the lime is as dry CAO, direct from the lime kilns, in small lumps, the idea being a saving

in lime and a quicker action of the lime upon the impurities in the juice. When slaked lime is used or lime moistened with "sweets" the addition causes a lowering in the temperature of the juices and hence a loss of heat. The amount of lime to be added plays a very important part in the liming of the juices. If too small an amount of lime is used, some of the impurities naturally remain in the juice, and the juice is liable to become acid, causing inversion of some of the sugar. If too much lime is used a lot of unnecessary time and a waste of $CO_2$ is the result in carbonation. We have found that adding 3% of the original weight of the beets, in lime works out well. In the extracting of sugar from the dried beets we figured as follows: Multiply the weight of the juice by the degrees brix (uncorrected); divide this by 15 and take 3% of the result. In adding the lime the juice should be agitated and the lime added in a flat layer.

The temperature at which the juices should be defecated is also an important item. At a temperature below 70° C., some of the lime is dissolved in the juice, the amount depending on the temperature. This lime forms a monosucrate of calcium and is difficult to decompose even at high temperatures. At 80° C. only about .25% lime is dissolved, and this shall amount is negligible. Temperatures about 80°-85° C. are not good for defecating, owing to the fact that some of the precipitated substances are likely to redissolve. Only when a bad lot of beets is to be worked is it advisable to use a higher temperature and a larger per cent of lime. So the working temperature is from 70° - 82° C. We have found

that if the juice is heated to 70° and the lime added and the temperature of the juice raised to 80° in about 15 minutes the best results will be obtained in defecating.

## First Carbonation.

After the juices have been treated with lime they must be treated with some re agent to precipitate this lime. Carbon dioxide is now used universally and has been found to better than any of the others which have been suggested. The carbonation is carried on in a tank whose height is about twice the diameter. The tank is fitted with perforated pipes for distributing the $CO_2$ and is heated from below. Steam coils cannot be used owing to the fact that they quickly become covered with a layer of lime and thus absorb a large amount of heat. No agitator is necessary as the passage of the $CO_2$ through the juice thoroughly churns up the latter. The height of the tank is very important as there is considerable foaming in carbonating, and this is quite likely to rise to a height of four to five feet above the surface of the juice. A small amount of oil or grease added to the juice prevents this foaming somewhat and in no manner affects the juice. It is best also to have a large exit pipe above the carbonating tank, through which the spent $CO_2$ may escape.

The process of carbonating is simple. The limed juice is run directly into the carbonation tank and the juice heated to 85° - 88° C. The CO is admitted slowly at first to prevent too much foaming, and then faster until carbonation is complete. Simple as the process may seem it requires the greatest watchfulness of the man in charge. Spoon

tests should be made every two or three minutes and when the break is noticed, alkalinity tests should be made until the right point is reached. When carbonation begins, the lime separates very slowly from the juice, but as it proceeds for some time a quick separation of the lime from the juice will be noticed. From then on, the greatest care should be taken to insure against over carbonation. Over carbonation will cause an inversion of some of the sugar and difficulties in filter-pressing. If carbonation has not been carried far enough the juice will be slimy and will quickly clog up the pores of the cloths in the filter press. The carbonation is complete when the alkalinity of the juice has reached .07 to .09, and to go below this is unsafe, as the sugar may invert if the juices are allowed to stand for any length of time. In regard to time, the juices should never be allowed to stand for any long period, as the juices become sour, even if previously limed and carbonated.

The chemical reactions occurring in the carbonation process are not fully understood. When $CO_2$ is added, the lime not only changes to carbonate, but a double salt of the same is formed, together with calcium sucrate and some caustic lime. This caustic lime forms a gelatinous precipitate which contains considerable sugar in the form of lime sucrates. All these compounds are partly decomposed by heat and dilution, and subsequent treatment with $CO_2$ breaking them up completely. It frequently happens, however, that small amounts of these compounds are not decomposed and remain in the scum, thus causing increased sugar loss.

Besides those reactions mentioned there are side reactions occurring, some of which are favorable and some unfavorable. A favorable reaction is the precipitation of those lime salts which are soluble in the alkaline juice, along with the calcium carbonate. Then there is the coagulation of the soft, slimy, inorganic or organic precipitates which were formed during defecation. The main unfavorable condition is the fact that some insoluble lime sucrate is retained by the precipitates or scums, but this is small and only occurs when unfavorable conditions ensue in liming and carbonating.

Carbonation of the juices should be completed in about fifteen minutes. Most of the troubles occurring in the process of carbonation are due to too slow a saturation of the juices. This is due to many causes. A lean gas may be used; the pumps may work badly; pipes may become clogged or too much lime may have been added in defecating. All these cause a lengthening of the duration of carbonating. The condition of the raw beets has a great deal to do with slow carbonation. Overripe or decayed, beets contain a lot of pectic substances which are extracted along with the sugar, and these appear to combine with the lime and thicken the juice.

## Treatment of Sludge.

After the first carbonation, the juice is pumped under a pressure of two or three atmospheres, to the filter-presses, and here all the precipitates are retained and form what is known as the "press cake." This press cake contains

about 40% solids and 60% juice, and this juice usually contains from 4-6% of sugar, depending on the amount of sugar in the defecated juice, and upon the amount of insoluble lime sucrate formed. Therefore it is necessary to run hot water through this cake and obtain this juice. This sweetening off should work so as to obtain the juice in as concentrated form as possible, and the least possible amount of water should be used.

The washing should be done until only about 1% of sugar remains in the cake; below the value it does not pay to work.

The diluted juice may be concentrated and added to the main bulk, or may be mixed with the lime used for defecation. The cake is then thrown away.

## Final Carbonation and Filtration.

The juice from the filter presses is now sent back to the tanks for a second carbonation. The juices in passing through the presses have cooled down considerably, and are therefore sent through exhaust-steam heaters which heat the juice up to 95-100° C. This second carbonation is to insure complete saturation of the juice and to bring the alkalinity down to the desired point. The high temperature used in this second carbonation is to allow the lime still retained in the juice to further act upon the non sugars, and to insure complete decomposition of any lime sucrate formed. The desired point of alkalinity has been the subject for much discussion, some authorities claiming that the juices should be neutral when sent to the concentrators. Classen claims that the juices should be alkaline to the extent of .03-05%, owing to the fact that alkalinity decreases during concentration, and that if the juices are neutral, they will become acid during the evaporation, and thus cause invert sugar. The best treatment is probably to carbonate the juices (second carbonation) down to an alkalinity of .05, filter press, and then treat the filtered juices at 95° C with sulphurous acid down to .02-.03. The sulphurous acid helps to precipitate the remaining impurities and gives a clear straw colored juice. The juice is then subjected to a final filter-pressing, some manufacturers even filter-pressing twice to insure a perfect filtration.

The juice is now ready for the concentrators.

## Concentrating and Boiling.

The proper working up of the juices in the evaporators depends a great deal upon their condition as they come from the presses. During evaporation the alkalinity (if any) decreases, and if it is too low at the start, the juices may become slightly acid and inversion takes place. If, however, they are of proper alkalinity, there is no inversion and in fact the juices are improved, for at the temperature at which evaporation is carried on, the alkalies act energetically on the non-sugars, such as albumens and amides, which are not thoroughly decomposed during defecation. This is under the conditions that the temperature does not rise above 115°-120° C, and that the alkalinity does not decrease to neutrality.

The advantages gained by having the juice slightly alkaline when sent to the evaporators more than offset those by having them neutral, or nearly so; for, although in the latter case, a clearer juice is obtained, inversion is very apt to take place and they become darker in color. The alkalinity required differs for different juices, depending upon the condition of the beets from which they were obtained, but the value usually lies between .05 and .1%.

The evaporators concentrate the juice up to a density of 55°- 60° Brix, removing about 80% of the water present. A higher concentration than 65° Brix is not economical as such a heavy syrup makes the vacuum pan work harder.

An average factory treats 550 tons of beets per day, yielding about 1,375,000# of thin juice to be concentrated. To evaporate such an enormous amount of water with

economy it is necessary that the most efficient and up-to-date methods be used. Of course, by using dried beets the amount of water to be evaporated will be diminished by about half, but that does not affect the principles involved. The heat-transference from steam to the boiling liquid in any evaporator is in proportion to:

1. - The speed of the movement of the liquor over the heating surface;

2. - Inversely as the height of ~~l~~iquor or the pressure on the heating surface;

3. - The speed with which the steam circulates over the heating surface;

4. - The speed and thoroughness with which the condensed water is taken away from the heating surface;

5. - The completeness of the vacuum in the evaporating chamber;

6. - The conductivity of the heating surface, that is, its freedom from scale and foreign substance;

7. - Inversely as the Viscosity of the liquor;

8. - The temperature of the liquor, which depends upon the pressure under which it boils;

9. - The magnitude of the temperature drop between the steam and the boiling liquor.

Multiple-effect evaporators (usually triple effect) which are made to satisfy the above conditions as nearly as possible are used for carrying on this part of the process.

The syrup coming from the last effect, having a density of 55°-60°Brix, is now heated to nearly 100°C

and carbonated to neutrality or .03-.04 alkalinity. It is now ready for the "strike" pan, where it is boiled to grain. This is an art which is acquired only by long practice, and a sugar boiler who has been used to one kind of apparatus may get bad results with a new kind.

Boiling in grain consists in forming the necessary number of crystals and then in further boiling allowing only these particular crystals to grow without forming a perceptible number of new ones. As there is no chemical or physical test which will determine when the desired point is reached, the operator accomplishes this by the outward appearance of the syrup and upon his judgment depend the yield as well as the proper and efficient working of the centrifugals.

In order for a sugar solution to deposit crystals it is necessary that it become supersaturated, and this is accomplished by evaporating to a smaller volume while at the temperature at which it is saturated. The crystals do not deposit immediately, the rapidity of growth increasing with the purity of the solution and the number of crystals already present. An impure solution requires a longer time as well as a stronger supersaturation to allow the crystals to grow.

It is essential in obtaining good crystals that the supersaturation, and consequently the temperature, should be the same at all points of the crystallizing mass and consequently the solution, must be stirred continually, especially when fresh syrup is added. The bubbles rising during boiling agitate it sufficiently during the intervals between

the additions of fresh syrup; however, in order that the fresh syrup be mixed and so not dissolve any crystals which have formed, it is sent in at the bottom in fine streams and at a higher temperature than that of the boiling mass. This causes a great number of steam bubbles to be suddenly generated and thus rapidly mixes the entering syrup with that already present.

The syrup in the pan is first taken down to such a point that it is supersaturated and then the formation of crystals is started by the shock produced by a sudden introduction of new syrup. When a sufficient number of crystals have formed the boiling is so conducted that no new ones will be formed, but these already formed will keep on growing. If the syrup is introduced periodically the coefficient of supersaturation is allowed to reach 1.2 and then enough syrup is added to bring it down nearly to 1.0. In no case should it be allowed to drop below 1.0 unless too many crystals have been formed. By the coefficient of supersaturation is meant the ratio of the amount of sugar on a definite amount of supersaturated solution to the amount in the same amount of saturated solution at the same temperature. After the last addition of syrup the supersaturation coefficient is brought up to the required amount, depending on the process of working up the "massecuite," and the partly crystallized mass is then sent to the crystallizers. In order to obtain good crystals it is necessary that the boiling be conducted for at least six or eight hours.

The treatment of the partly crystallized mass after it reaches the crystallizers depends upon whether it is a stiff or a thin "massecuite." If stiff it must be kept at the temperature at which it leaves the vacuum pan for some time in order that new crystals may not form; if it is thin, however, it can be cooled more quickly. After cooling to about 55° C, which takes from 18 to 20 hours, during which time it has been slowly stirred, enough dilute sirup is added to make the mass sufficiently fluid to go to the centrifugals.

The working of these centrifugals depends upon the character of the crystals; if there are many fine ones they will form a film, and it will be hard to remove the molasses; if they are of the correct size they will not pack so tightly but what the molasses is thrown out quite easily. The massecuite is run into the centrifugals while they are stationary, and after as much of the sirup as possible has been thrown out, the layer of crystals is washed with water. The sugar is then removed, being in a warm and somewhat damp condition. In order to make the crystals of about uniform size, and to dry them, the sugar is sent through a long rotating cylinder, which is heated. The crystals are carried to the top and dropped down periodically, and meanwhile progress from one end of the cylinder to the other. On discharging, the crystals are of nearly the same size and are ready for storing or shipping.

The molasses coming from the centrifugals for the first time is usually sent back and retreated with the thin juices in order to obtain a better yield of sugar. That

which is not sufficiently pure in sugar is not sent back, but is mixed with the exhausted beets, which have been dried, pressed, and sold as cattle food.

| Ob. Brix | Temp. | Corr. Brix. | Polar | Purity | Increase |
|---|---|---|---|---|---|
| 25.1 | 23 | 25.5 | 21.4 | 83.9 | -1.6 |

### After Sulphuring.

| Juice | Alkalinity |
|---|---|
| 35# | .015% |

| Ob. Brix | Temp. | Corr. Brix. | Polar | Purity | Increase |
|---|---|---|---|---|---|
| 27.7 | 23.5 | 28.1 | 23.3 | 82.9 | -1. |

### Juice going to pan.

| Wt. | Brix at 20° | Polar | Calc. Sugar | Alkalinity |
|---|---|---|---|---|
| 35# | 28.1 | 23.3 | 8.15# | .04 |

### In Pans

| Vaccum - Cm.Hg. | Boiling Point °C. |
|---|---|
| 20 cm. | 90° |

### Coming from Pans.

| Time of Cooling | Wt. of Massecuite | Brix. |
|---|---|---|
| 4 hrs. | 11.3# | 87° |

### After Centrifugating.

| Wt. of Sugar | Purity |
|---|---|
| 3# | 98% |

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### Sweet Waters

| Before Concentrating | | After Concentrating | |
|---|---|---|---|
| Wt. | Brix. | Wt. | Brix. |
| 22# | 6° | 6# | 22° |

### Lime Cake from Filter Press.

| Wt. Cake | % Sugar | Sugar loss in Cake |
|---|---|---|
| 8.5# | 1% | .085 |

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### 74# Juice from Diffusor.

### Before First Liming

| Obs. Brix | Temp. | Corr. Brix. | Polar. | Purity |
|---|---|---|---|---|
| 17.1 | 22.5 | 17.45 | 14.1 | 80.9 |

### First Liming.

| Lime Used | Time for liming | Temp. start | Temp. finish |
|---|---|---|---|
| 2.5# | 15 min. | 72° C. | 82° C |

### First Carbonation

| Time for Carbonating | Temp. start | Temp. finish |
|---|---|---|
| 35 min. | 82° C. | 80° C |

### After First Carbonation

| Juice | Alkalinity |
|---|---|
| 61# | .07 |

| Obs. Brix. | Temp. | Corr. Brix. | Polar | Purity | Increase |
|---|---|---|---|---|---|
| 17.25 | 27° | 17.88 | 15.2 | 85 | 4.1 |

### Second Liming.

| Lime used | Time for liming | Temp. start | Temp. finish |
|---|---|---|---|
| .31# | 10 min. | 90° C. | 93° C |

### Second Carbonation

| Time for Carbonating | Temp. start | Temp. finish |
|---|---|---|
| 15 min. | 92° C. | 90° C. |

### After Second Carbonation

| Juice | Alkalinity |
|---|---|
| 51.5# | .05 |

| Obs. Brix | Temp. | Corr. Brix | Polar | Purity | Increase |
|---|---|---|---|---|---|
| 21.75 | 23° C | 22.15 | 19.2 | 86.7 | 1.7 |

After Sulphuring

| Juice | Alkalinity |
|---|---|
| 47# | .025 |

| Obs. Brix. | Temp. | Corr. Brix. | Polar | Purity | Increase |
|---|---|---|---|---|---|
| 24.25 | 23.4 | 24.65 | 21.5 | 87.3 | .6 |

Juice going to Pans

| Wt. | Brix. | Polar. | Calc. Sugar | Alkalinity |
|---|---|---|---|---|
| 47# | 24.24 | 21.1 | 9.91 | .025% |

Juice in Pans.

| Vacuum cm. Hg. | Boiling Point |
|---|---|
| 30 cm. | 85° C. |

Coming from Pans

| Time of Cooling | Wt. of Massecuite | Brix. |
|---|---|---|
| 4 hrs. | 12.42 | 91° |

After Centrifugating

| Wt. Sugar | Purity |
|---|---|
| 4# | 98% |

Lime Cake from Filter Press.

| Wt. Cake | % Sugar | Sugar loss in Cake |
|---|---|---|
| 10# | 6% | .6# |

## Discussion of Data.

A great deal of time and labor was lost in carrying on this work, due to the fact that crude and imperfect apparatus was used in the treatment of the juices coming from the diffusors. The liming tank was simply a small cylindrical sheet iron tank, about 2-1/2 ft. in diameter by 16" deep. The tank was heated from beneath by two gas burners and considerable time was lost in heating up the juices and keeping them at the right temperature. The same tank was used for carbonating, an improvised 1/4" perforated pipe connected to a $CO_2$ tank was simply placed in the bottom and the juice carbonated. The tank was by far too shallow for carbonating, the rapid pasage of $CO_2$ through the juices causing considerable loss in frothing and waste of the gas. If the $CO_2$ was passed in slowly too long a time was taken to complete the carbonation. It will be seen from the data that from 35 min. to 1 hour and a half was required, while the correct length of time for carbonating is 15 min.

Although a great many runs were made previous to these two set herein-with, these latter were the only ones on which complete data was obtained. Due to imperfections in the apparatus used and unavoidable interruptions and delays, the results obtained from these other numerous runs are hardly worth while embodying in this thesis, with the exception of the dataobtained in diffusion.

It will be noticed in the first run that there was a decrease in purity after the second carbonation and after

sulphuring. After the first carbonation the purity increased as it should, the probable reason for the decrease being that the juice stood over night between the first and second carbonation and some inversion took place. In the second run there was a steady increase in the purity all the way through, these juices being treated immediately and taken through the process without delay.

Difficulties arose in the concentrating of the juices owing to the fact that both concentrating and sugar boiling had to be carried on in the same apparatus, this being a single effect, and to the fact that the pan was not of sufficient capacity nor of correct design for this work. The pan being spherical, frothing could hardly be prevented even with the addition of soap or butter, and considerable loss was sustained because of this. Then again, there was no means of examining the syrup so as to determine the size and number of sugar crystals being formed. Another source of trouble was in obtaining a vacuum sufficiently high to prevent caramelizing of the sugar. Due to these difficulties the crystals formed were small and were not as large as are obtained in sugar factories. This caused trouble in the centrifugal by packing too tightly and thus hindering quick separation of the molasses and crystals, and also subsequent washing of the remaining molasses from the crystals.